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I, LEANNE MYNOTT, MANAGER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2003906074 for a patent by COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION as filed on 03 November 2003.

WITNESS my hand this
Sixteenth day of November 2004

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Patents Act 1990

PROVISIONAL SPECIFICATION

Invention Title: **Packaged food product and process**

The invention is described in the following statement:

Packaged Food Product and Process

Field of the invention

The present invention relates to a process for producing a packaged food product and to a packaged food product.

Background of the invention

A number of processes have been proposed to produce packaged food products that exhibit desired organoleptic and nutritional qualities, have extended shelf life and have suppressed growth of bacteria, yeasts and moulds.

Early packaged food products were typically canned food. In these products, the food was heated to high temperature (either before or after canning) and then sealed in a can. Exposure to high temperature killed the microorganisms present in the food and the subsequent sealing of the food in the can prevented ingress of further microorganisms. Canning of food products is widely practiced to this day. However, canning processes are not suitable for packaging all foods as some foods are detrimentally affected by the high temperature used in canning operations.

In the early 1990's, the first commercial use of ultra-high pressure treatments of foods took place. In these processes, the food was subjected to high pressure, typically in excess of 400 MPa. Ultra-high pressure treatment of foods is known to be effective in inactivating vegetative bacteria, yeasts and moulds. However, ultra-high pressure treatment has not been found to be effective at destroying bacterial spores, denaturing enzymes or destroying ascospores of heat resistant moulds.

In order to overcome the limitations of ultra-high pressure treatments, US patent no. 6,086,936 described a process where a combination of ultra-high pressure and elevated temperature is used to treat food. Applying ultra-high pressure to the food was described as causing an instantaneous adiabatic temperature change. It is stated in this patent that the ultra-high pressure and temperature combination contribute synergistically to the lethality of the process. This patent states that the elevated temperature should be greater than ambient (20 C), more preferably greater than 75 C. Preferably, the pre-pressurised temperature is less than 105 C. The ultra-high pressure is stated to be greater

than 75,000 psi (516 MPa), and preferably less than about 250,000 psi (1,722 MPa). The claims of this patent are limited to treating a non-dairy food product having a pH equal to or greater than 4.6.

A wide variety of foods and other materials are susceptible to loss in quality during storage under atmospheric levels of oxygen. The damage can arise from chemical oxidation of the product, from microbial growth, and from attack by vermin – much of which may be avoided by reducing the oxygen availability in the environment of the materials. In the field of packaging, relatively low-oxygen atmospheres have traditionally been generated by vacuum packing and inert gas flushing. Such methods are not, however, generally applicable for various reasons. For example:

- soft porous foods such as cakes cannot be subjected to strong vacuum;
- fast filling speeds generally preclude substantial evacuation of or thorough inert gas flushing of food packages;
- filling some gas-flushed containers, such as beer bottles often results in occlusions of air;
- evacuation or flushing offers no residual capacity for removal of oxygen, which may have desorbed from the food or entered the package by leakage or permeation.

As a consequence there has been much interest in chemical techniques for generating low-oxygen atmospheres and deoxygenating liquid or semi-liquid foods. Thus, there are approaches based on the use of oxidisable solids, for example porous sachets containing iron powder. In another technique, oxidisable MXD-6 Nylon is blended with polyester in the walls of flow-moulded containers. The effectiveness of this depends on the presence of a cobalt salt catalyst, moreover the speed of oxygen removal is limited by the oxygen permeability of the polyester. Further methods include sandwiching crystalline oxidisable material between the layers of multilayer containers, and including a catalyst for the reaction of oxygen with hydrogen in a sandwich arrangement as above or as a deposit on the inner surface of the package.

Another method which might be used to provide oxygen scavenging in packages as required, is disclosed in Rooney, M.L., Chemistry and Industry, 20 March 1982, pp.197-198. This method involves the inclusion of a photo-oxidizable rubber and a photosensitising dye into a polymer film packaging material and then exposing it to visible light. Similar methods are disclosed in Rooney, M.L. and Holland, R.V.., Chemistry and Industry, 15 December 1979, pp.900-901 and Rooney, M.L., Journal of Food Science, Vol. 47, No.1, pp.291-2294, 298. These methods initiate oxygen scavenging upon illumination and require constant illumination of the package in order to maintain the scavenging effect. US Patent No. 5211875 proposes an alternative method intended to avoid the problem of oxygen-sensitivity prior to use, involving an oxidisable organic compound (typically 1, 2-Polybutadiene) and a transition metal catalyst (typically cobalt salt). Oxygen scavenging is initiated by exposing the composition to an electron beam, or ultraviolet or visible light.

Australian patent no. 672 661, the entire contents of which are herein incorporated by cross-reference, describes a solid phase composition for reducing the concentration of ground state molecular oxygen present in an atmosphere or liquid comprising at least one reducible organic compound which is reduced when the composition is subjected to predetermined conditions, the reduced form being oxidisable by ground state molecular oxygen, wherein the reduction and/or subsequent oxidation of the organic compound occurs independent of both constant illumination with visible light and the presence of a transition metal catalyst.

The solid phase composition of Australian patent no. 672 661 allows the oxygen scavenging ability to be activated when desired by the user by exposing the composition to the predetermined conditions to reduce the organic compound to an oxidisable exposure to light of a certain intensity or wavelength, by the application of heat, γ-irradiation, corona discharge or an electron beam. The organic compound may be reduced by incorporating in the composition a reducing agent which in turn can be activated under predetermined conditions, say, by heating.

The composition described in Australian patent no. 672 661 may be provided in the form of a packaging film or laminate.

Australian patent no. 672 661 also describes a method for reducing the concentration of molecular oxygen present in an atmosphere or liquid by exposing the atmosphere or liquid to the composition and reducing the reducible organic compound, or by exposing the atmosphere or liquid to a pre-reduced form of the composition.

Summary of the invention

In a first aspect, the present invention provides a method for producing a packaged food product including the steps of adding a food product to a package and subjecting the food product to an ultra-high pressure treatment, wherein the food product is in an oxygen-scavenging environment either before or after the ultra-high pressure treatment.

In one embodiment, the food product is subjected to the ultra-high pressure treatment prior to being added to the package. In an alternative embodiment, the food product is placed in the package and subsequently subjected to the ultra-high pressure treatment.

The food product may be placed in an oxygen scavenging environment by any suitable method. For example, the food product may be placed in close proximity to, or in contact with, an oxygen scavenging material. Alternatively, an oxygen scavenging compound, such as an enzyme or a chemical compound, may be mixed with the food product, or the food product may be placed in an oxygen scavenging package.

It is preferred that the food product is placed in packaging that includes an oxygen scavenging material.

The oxygen scavenging material is suitably an oxygen scavenging packaging material.

The oxygen scavenging material is most suitably the solid composition described in Australian patent no. 672 661. Using this oxygen scavenging material allows the oxygen scavenging to be initiated at a time of choosing of the user. The oxygen scavenging may be initiated before the food is added to the package or it may be initiated after the food is added to the package. In embodiments where the food product and the package are subjected to the ultra-high pressure treatment, the oxygen scavenging may be

initiated before the ultra-high pressure treatment or after the ultra-high pressure treatment.

In an especially preferred embodiment of the present invention, the food product is placed in a package that includes an oxygen scavenging material, and the food and package are subjected to the ultra-high pressure treatment.

Most preferably, the oxygen-scavenging environment is maintained after the ultra high pressure treatment has been completed.

The ultra-high pressure treatment step may involve subjecting the food product to a pressure in excess of 200 MPa, preferably to a pressure in the range of from 400 MPa to 1500 MPa. In the more preferred embodiment, the food is subjected to a pressure in the range of from 500 MPa to 1200 MPa, even more preferably from 600-800 MPa.

The temperature used in this process is not especially critical. The temperature at the time of high pressure treatment may range from 0 to 75 C, more preferably from ambient temperature to 60 C. The invention is preferably practiced without any additional heating provided during the high pressure processing step.

The food product treated by the method of the present invention is suitably a high acid food. The food preferably has a pH of less than 4.6. The food may be a fruit-based food product.

The present inventors have surprisingly found that treating food in accordance with the method of the present invention produces a packaged food product that is commercially sterile, has long shelf life, has reduced tendency to discolour and shows diminished or no growth of yeast, bacteria or heat resistant moulds. This is an unexpected outcome as it is believed that ultra-high pressure treatment by itself is not sufficient to disrupt the ascospores of heat resistant moulds to an extent that prevents germination and growth of the heat resistant moulds. Thus, the combination of ultra-high pressure treatment and packaging in an oxygen-scavenging environment provides an unexpected, synergistic result.

Without wishing to be bound by theory, the present inventors also believe that, in preferred forms of the invention, conducting the high pressure processing in the oxygen

scavenging environment may increase the rate of oxygen scavenging, which may also assist in increasing the inactivation of the microbes and moulds. The increased rate of oxygen scavenging may also aid in reducing the effects of oxidation, such as of browning of the food and the loss of nutrients, flavour and aroma.

It is preferred that the packaging used in the present invention provides a barrier to oxygen permeability. Suitably, the packaging used in the present invention includes a laminate having one layer with an oxygen scavenging ability and another layer that provides a barrier to oxygen entering the packaging from the external atmosphere.

In a second aspect, the present invention provides a packaged food product in which a food product is packaged in a package and the package subsequently sealed, characterised in that the food product is subjected to an ultra-high pressure treatment and the food product is in an oxygen-scavenging environment in the package.

In a third aspect, the present invention encompasses a food product produced by the method of the first aspect of the present invention.

In all aspects of the present invention, the food product may have one or more materials for treating spores, particularly ascospores, added thereto. This one or more materials may be an enzyme, such as chitinase.

Examples

Example 1:

This example demonstrates the high pressure treatment of heat resistant moulds in three different packaging films

High pressure (HP) inactivation was carried out in a 2L high pressure processing (HPP) unit. Spores were packed in 3 types of packaging film, as specified below:

Material and methods

Cultures: Two heat resistant mould species, Byssochlamys fulva FRR 3792, from heat processed strawberry purée and Neosartorya fischeri FRR 4595 from heated strawberry purée, were used.

Packaging films: Three packaging films with varying oxygen permeabilities were included in the test: OPET/EVOH/oxygen scavenging material/CPP, hereafter referred to as "oxygen scavenging film", EVA, very permeable to O_2 and a heat sealable laminate containing EVOH (low permeability to O_2 and commonly used as an oxygen barrier film). (OPET = oriented polyester, EVOH = ethylene vinyl alcohol, CPP = cast polypropylene, EVA = ethylene vinyl acetate).

Procedure:

Fungi were grown on MEA at 30°C for 3 weeks and spore suspensions (10^4cfu/mL) were prepared in 20 °Brix syrup with citric acid, pH 4.2. Spore suspensions of B. fulva and N. fischeri were blanched at 95°C for 5 minutes. Five mL of blanched sample was poured into15 pouches (5 x 10 cm) of each film (oxygen scavenging film, EVA and EVOH laminate) for each fungal species. These pouches were then HP treated at 600 MPA for 0 (control), 60 and 120 seconds at ambient temperature (approx. 25°C), 5 pouches of each packaging film per treatment for each mould species. After HPP, the treated pouches were incubated for 2 weeks at 30°C, then checked for visible mould growth.

Results:

After 2 weeks incubation at 30°C, growth was observed in all EVA and EVOH laminate pouches that had not received a pressure treatment (control pouches). No visible growth was observed in the oxygen scavenging film control pouches.

Similarly, for pouches that had received the HP treatment of 600 MPa for 60 and 120 sec., growth was observed in 5/5 (all) EVA and EVOH laminate pouches for both fungi at both pressure treatment times, whereas no visible growth was apparent in the oxygen scavenging film pouches.

Inoculum viability test

After 2 weeks incubation, pouches containing N. fischeri and B. fulva in oxygen scavenging film packages were opened and 0.1 mL samples were plated out onto duplicate plates of DRBC and MEA to check the inoculum viability. All plates were incubated at 30°C for 5 days. The results are as follows:

Table 1: Oxygen scavenging film pouches

Treatment	B. fulva cfu/mL	N. fischeri cfu/mL
0 seconds	2.6×10^2	8.4×10^2
60 seconds	5.3×10^1	5×10^1
120 seconds	1×10^1	2×10^2

Discussion:

There appears to have been a reduction in numbers of both B. fulva and N. fischeri in all pressure treatments (including the control) in the oxygen scavenging film packaging films. Without wishing to be bound by theory, it is postulated that the very low oxygen environment may be detrimental to the spores, particularly those that have been pressure treated and may be sub-lethally injured. The low numbers recovered after 2 weeks incubation indicates that although some ascospores have survived the pressure treatment, they are prevented from growing in the oxygen scavenging film packaging system. This can be compared with results for the other two types of film (EVA, EVOH laminate) where visible growth of both fungi after both pressure treatments was observed after 2 weeks incubation at 30°C.

Example 2

This example investigated high pressure treatment of yeasts in three different packaging films

High pressure inactivation was carried out in 2L high pressure processing (HPP) unit. Yeasts were packaged in 3 types of packaging film.

Materials and methods

Cultures: Two yeasts, Saccharomyces cerevisiae FRR 1813 from beer and Pichia anomala FRR 5220 from fermenting vanilla blueberry yoghurt were used in this example.

Packaging material:

Three packaging films with varying oxygen permeabilities were included in the test: OPET/EVOH/oxygen scavenging material/CPP, hereafter referred to as "oxygen scavenging film", EVA, very permeable to O_2 and a heat sealable laminate containing EVOH (low permeability to O_2 and commonly used as an oxygen barrier film). (OPET = oriented polyester, EVOH = ethylene vinyl alcohol, CPP = cast polypropylene, EVA = ethylene vinyl acetate).

Procedure:

Yeasts were grown on MEA at 25°C for 3 weeks and cells suspensions (10^4cfu/mL) were prepared in 20 °Brix syrup with citric acid, pH 4.2. Five mL of cell suspension was poured into 15 oxygen scavenging film, 15 EVA and 15 EVOH pouches (5 x 10 cm) for each yeast. These pouches were then HP treated at 400 MPa for 0, 60 and 120 seconds at 25°C. After high pressure treatment one pack/pouch from each group was opened and 0.1 mL was plated out onto duplicate TGY plates and incubated 3 days at 25°C. Rest of sample pouches were incubated for 3 weeks at 25°C

Results:

Immediately after pressure treatment, the plate count results were as follows:

Table2:

Treatment	S. cerevisiae cfu/mL			P. anomala cfu/mL		
	EVA	EVOH laminate	oxygen scavenging film	EVA	EVOH laminate	oxygen scavenging film
60 seconds	No growth	No growth	No growth	2.6×10^2	3.7×10^2	2.7×10^2
120 seconds	No growth	No growth	No growth	No growth	No growth	No growth

After 3 weeks incubation sample pouches were checked for visible yeast growth. Growth was not visible macroscopically, so one pouch from each sample was opened and

enumerated by plating onto duplicate TGY plates which were incubated 3 days at 25°C, with the following results.

Table 3:

Treatment	S. cerevisiae cfu/mL			P. anomala cfu/mL		
	EVA	EVOH laminate	oxygen scavenging film	EVA	EVOH laminate	oxygen scavenging film
Control (0 Sec)	2.5×10^5	2.8×10^5	1.0×10^5	2.5×10^6	3.3×10^6	6.2×10^5
60 seconds	No growth	No growth	No growth	$>10^5$	$>10^5$	$>10^5$
120 seconds	No growth	No growth	No growth	No growth	No growth	No growth

Discussion:

Pressure treatment at 400 MPa for 120 sec inactivated both species of yeast. P. anomala was slightly more resistant, with some cells surviving 60 sec at 400 MPa, conditions under which S. cerevisiae was inactivated. Packaging films did not appear to greatly affect the growth of the yeasts, although growth in the non-HP treated packs of oxygen scavenging film was slightly lower than the other two types of packaging film. Both yeast species are fermentative, so they would be expected to grow at very low oxygen levels, or even anaerobically. The lack of visible growth and fermentation in the untreated packs could be attributed to nitrogen limitation of the suspension medium (sucrose and citric acid).

Example 3

This example demonstrates the effect of HPP on an experimental oxygen scavenging technology packaging material

Eight OPET/EVOH/oxygen scavenging material/CPP (hereafter referred to as "oxygen scavenging film") pouches (with a 0.0288 m2 surface area) were aseptically filled with 50 ml of aerated distilled water (,OPET = oriented polyester, EVOH = ethylene vinyl alcohol, CPP = cast polypropylene).

Each pouch contained an oxygen scavenging component.

HPP for two minutes at 600 MPa was then applied to four OPET/EVOH/oxygen scavenging material/CPP pouches. All eight pouches were then stored at 25 C.

The dissolved oxygen in the water of each pouch was analysed using a calibrated MI-730 Micro-Oxygen Electrode (Microelectrode Inc, USA) and OM-4 Oxygen Meter (Microelectrodes Inc, USA), at 25 C. Measurements were taken over time to monitor the rate of oxygen scavenging due to the oxygen scavenger contained in the pouches. Results are displayed in Figure 1.

Discussion – Example 3

The oxygen scavenging pouch exhibited a difference in the rate of oxygen scavenging (in water) during HPP; the effect was seen within an hour of HPP (Figure 1). The oxygen scavenging pouches exhibited an increase in the rate of oxygen scavenging during HPP (Figure 1). A larger decrease in the oxygen content, which was maintained over time, was seen in the oxygen scavenging pouches that had undergone HPP compared to the oxygen scavenging pouches that had not undergone HPP (Figure 1).

It was found that, during HPP the rate of oxygen scavenging of an oxygen scavenging film increased.

Those skilled in the art will appreciate that the present invention may be susceptible to variations and modifications other than those specifically described. It is to be understood that the present invention encompasses all such variations and modifications that fall within its spirit and scope.

Dated this 3rd day of November 2003

Commonwealth Scientific and Industrial Research Organisation

Australian Food Industry Science Centre

by its attorneys

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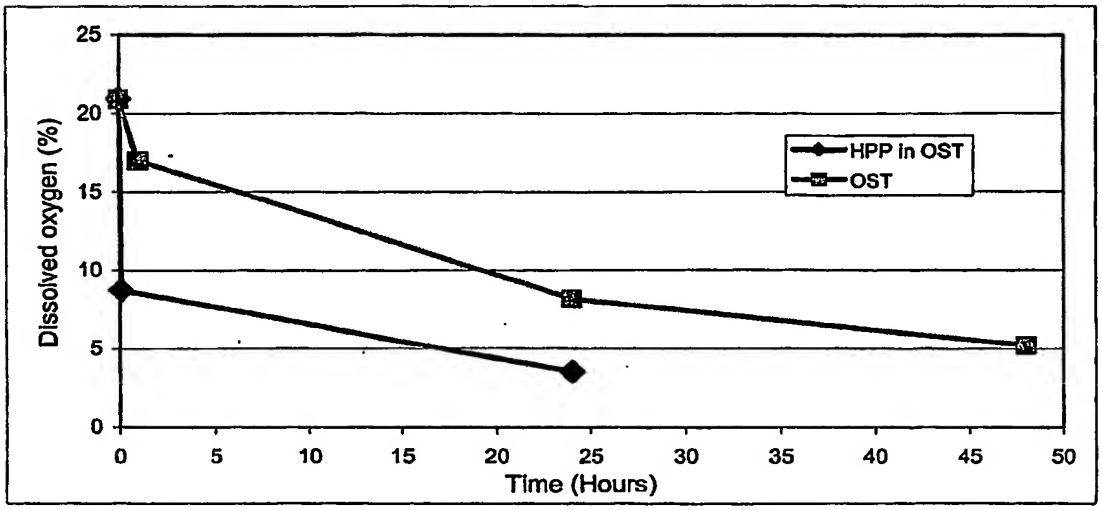

Figure 1 Effects of HPP on an oxygen scavenging pouch, at 25°C

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